Corrosion inhibitor for the chemical decontamination of primary coolant systems of nuclear power plants

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ABSTRACT

Radiation field reduction in the primary heat transport (PHT) system of water-cooled reactors can be achieved by 'dilute chemical decontamination (DCD)' process. The corrosion behaviour of carbon steel has been studied in the absence and presence of different concentrations of cinnamaldehyde in 4.76 mM L^{-1} of citric acid for different immersion times (6 h and 24 h) at 30 °C using weight loss, potentiodynamic polarization and impedance measurements. Cinnamaldehyde has given an inhibition efficiency of about 90% at 15.15 mM L^{-1} and inhibition efficiency is decreased to 82% when duration of immersion increased from 6 h to 24 h. Electrochemical studies follow the same trend as those observed in weight loss measurements. The formation of the adsorbed inhibitor film on the carbon steel was further confirmed by FTIR studies.

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1. Introduction

Carbon steel is widely employed as a material of construction in primary heat transport systems of heavy water reactor systems. At high temperature due to Schikorr reaction, magnetite (Fe_3O_4) is formed on the iron surface. The presence of these oxide deposits causes several difficulties such as poor heat-transfer efficiency and incorporation of the activated corrosion products which build up the radiation fields on these surfaces. To overcome these difficulties, chemical decontamination of the system is carried out. Chemical decontamination involves dissolution of the deposits on the surface using a specific chemical acid formulation [1]. Therefore, many different chemical decontamination methods have been developed [2–9].

In the last few years, a considerable amount of research on the chemical cleaning of nuclear steam generators has been performed in many laboratories. Among other aspects studied, the corrosion mechanisms involved in the use of the cleaning solvents [10–14] and the development of process applications [15–21] have been extensively explored. For this purpose, experiments were conducted on a number of sulphur free organic corrosion inhibitors and surfactants in a solvent based EDTA, citric acid and hydrazine which was originally developed for cleaning purposes at Ontario's Nuclear Generating Station [18].

The dilute chemical cleaning formulations contain an organic acid (which is a source of H$^+$ ions), a complexing agent to chelate Fe^{2+} ions and a reducing agent. Citric acid is one of the commonly used organic acids along with different complexing agents [22,23]. The addition of inhibitors is essential for further lowering of corrosion rate. The selection of inhibitors is based on the criteria of their effectiveness at low concentrations, less toxicity, thermal stability and less deleterious effect on the equipments used. Sulphur containing compounds cannot be used as they can cause stress corrosion cracking in stainless steel components which may be a part of the circuit. Among the class of compounds, the amines are most effective. Further, the aromatic amines are, in general, more efficient as compared to the aliphatic amines at a lesser concentration [24]. Investigations have shown acridine to be an effective inhibitor for acid corrosion of aluminium base alloys [25–27], copper [28] and iron [29]. Hoar and Khera [30] have studied the inhibition characteristics of diethanolamine for the corrosion of mild steel in acid medium.

Acridine, p-aminobenzaldehyde and diethanolamine have been used as corrosion inhibitors for carbon steel in citric acid [31]. A dilute chemical decontamination (DCD) formulation containing EDTA, ascorbic acid, citric acid (EAC) and Rodine-92B was reported

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Table 1
Corrosion rate and pH of carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde for an immersion period of 6 h at 30 °C

Concentration of inhibitor (mM L^{-1})	pH		Cinnamaldehyde	
	Initial	After 6 h	Corrosion rate (mmpy)	Inhibition efficiency (%)
Blank	2.67	2.81	1.30	–
0.76	2.68	2.79	0.82	36.9
3.78	2.67	2.75	0.39	70.0
7.57	2.65	2.72	0.37	71.5
15.15	2.64	2.66	0.13	90.0

for carbon steel [32]. A soft organic cleaning solution consisting of 3.5×10^{-2} M picolinic acid and 1.0×10^{-2} M ascorbic acid has been shown to be very effective in dissolving magnetite powder [33]. Once the oxide deposit dissolves, the cleaning solution attacks the base metal. Although the rates are low, the magnitudes are nevertheless significant and hence require addition of inhibitors. Electrochemical impedance and weight loss measurements have shown 5-hexyn-1-ol to be very effective in inhibiting the corrosion of carbon steel in the mixture of 3.5×10^{-2} M picolinic acid and 1.0×10^{-2} M ascorbic acid. In the light of several studies reported on the above topic, among three acids the corrosion rate increases in the following order [34].

Ascorbic acid < EDTA < Citric acid

Hence in the present work, the corrosion behaviour of carbon steel in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde and for different immersion time (6 h and 24 h) at 30 °C has been evaluated using weight loss, potentiodynamic polarization and impedance measurements to develop suitable corrosion inhibitor system for carbon steel in decontamination processes.

2. Experimental details

2.1. Weight loss measurements

Weight loss measurements were carried out for the carbon steel specimens of size 5 cm × 1 cm × 0.1 cm in 4.76 mM L^{-1} of citric acid containing different concentrations (0.76 mM L^{-1}, 3.78 mM L^{-1}, 7.57 mM L^{-1}, 15.15 mM L^{-1} and 37.85 mM L^{-1}) of cinnamaldehyde at 30 °C for 6 h and 24 h. The initial pH and final pH values of the test solution were also noted. After 6 h and 24 h, the carbon steel specimens were taken out, dried and final weights were noted. The corrosion rate CR (mmpy) and inhibition efficiencies were calculated using the formula,

$$CR(mmpy) = \frac{87.6 \times W}{ATD} \tag{1}$$

where W is the weight loss (mg), A is the area (cm^2), T is the time of exposure (h) and D is the density in (g cm^{-3}).

$$Inhibition\ efficiency\ (\%) = \frac{W_B - W_I}{W_B} \times 100 \tag{2}$$

where W_B and W_I are weight loss per unit time in the absence and presence of inhibitors.

2.2. Potentiodynamic polarization measurements

Potentiodynamic polarization measurements were carried out using SOLARTRON Electrochemical Measurement Unit (Model 1280B) at 30 °C for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of

cinnamaldehyde. Measurements were done initially and after 6 h and 24 h of immersion in the test solution at 30 °C. The area of the working electrode exposed was 1 cm^2 and the remaining portion was covered with red lacquer. A saturated calomel electrode and platinum electrode were used as reference and counter electrode, respectively. The initial pH was noted and the working electrode was immersed in a test solution for approximately 10–15 min for the attainment of steady state equilibrium. The polarization parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}) and Tafel slopes (b_a and b_c) were evaluated from potentiodynamic polarization measurements. The corrosion rate (mmpy) is calculated using the formula,

$$CR(mmpy) = 3.2 \times I_{corr}\ (mA\ cm^{-2}) \times \frac{Equivalent\ weight}{Density} \tag{3}$$

The inhibitor efficiency was calculated using the following formula:

$$IE(\%) = \frac{I_{corr} - I_{corr}^*}{I_{corr}} \times 100 \tag{4}$$

where I_{corr} and I_{corr}^* are corrosion current in the absence and presence of inhibitors.

2.3. Electrochemical impedance spectroscopy (EIS)

Impedance measurements were carried out using the same instrument as mentioned in potentiodynamic polarization measurements. The studies were carried out over a frequency range of 10 kHz to 10 mHz. Measurements were done initially and after 6 h and 24 h of immersion in the test solution. The impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) values were evaluated from Nyquist plots.

The percentage of inhibition efficiency was calculated using the following equation

$$IE(\%) = \frac{R_t^* - R_t}{R_t^*} \times 100 \tag{5}$$

where R_t^* and R_t are the charge transfer resistance in the presence and absence of inhibitors.

2.4. Atomic absorption spectrophotometric studies

Atomic absorption spectrophotometer (Spectraa 220, Australia) was used for estimating the amount of dissolved iron in the corrodent solution containing various concentrations of cinnamaldehyde in 4.76 mM L^{-1} of citric acid after exposing the carbon steel specimen for 6 h and 24 h. From the amount of dissolved iron, the inhibition efficiency was calculated.

$$Percentage\ inhibition\ efficiency = \frac{B - A}{B} \times 100 \tag{6}$$

where A is the amount of dissolved iron in presence of inhibitor and B is the amount of dissolved iron in absence of inhibitor.

2.5. Surface examination studies analysis of FTIR spectra

The FTIR spectra were recorded using Nicolet-380 FT-IR spectrophotometer for the carbon steel surface immersed in 4.76 mM L^{-1} of citric acid in the absence and presence of the inhibitor.

Table 2
Corrosion rate and pH of carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde for an immersion period of 24 h at 30 °C

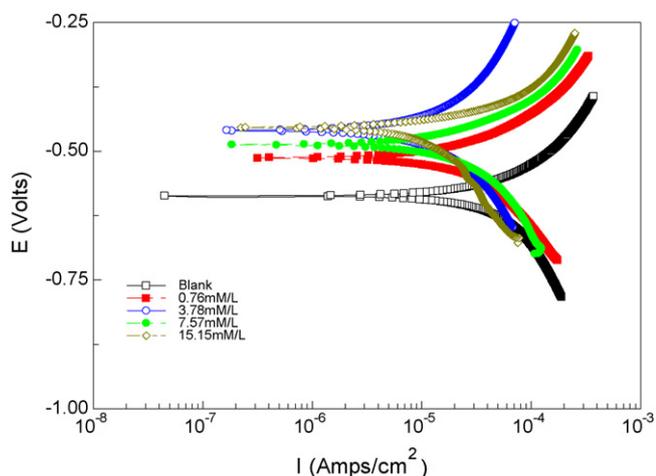| Concentration of inhibitor (mM L^{-1})	pH		Cinnamaldehyde	
	Initial	After 24 h	Corrosion rate (mmpy)	Inhibition efficiency (%)
Blank	2.65	3.13	0.45	–
0.76	2.63	2.95	0.34	24.4
3.78	2.62	2.75	0.19	57.8
7.57	2.61	2.70	0.13	71.1
15.15	2.61	2.69	0.08	82.2

Fig. 1. Potentiodynamic polarization curves for steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde (initial).
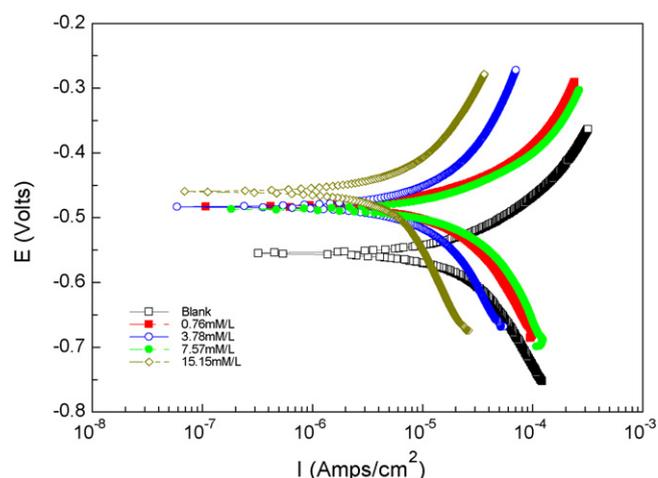
3. Results and discussion

3.1. Weight loss measurements

The percentage of inhibition efficiency and corrosion rate obtained from weight loss method at different concentrations of inhibitor for the corrosion of carbon steel in 4.76 mM L^{-1} of citric acid for an immersion period of 6 h and 24 h at 30 °C are given in Tables 1 and 2. From the table it can be seen that the inhibition efficiency is increased with increase in concentration of inhibitor from 0.76 mM L^{-1} to 15.15 mM L^{-1} Cinnamaldehyde gave an inhibition efficiency of about 90% at 15.15 mM L^{-1} (Table 1) and inhibition efficiency is decreased to 82.2% (Table 2) when duration of immersion increased from 6 h to 24 h. The pH values before and after the experiments were also noted and given in Tables 1 and 2. It can be seen that there is not much variation in pH values.

3.2. Electrochemical methods

3.2.1. Potentiodynamic polarization studies

Figs. 1–3 show the polarisation curves for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde obtained initially and for 6 h, 24 h of immersion
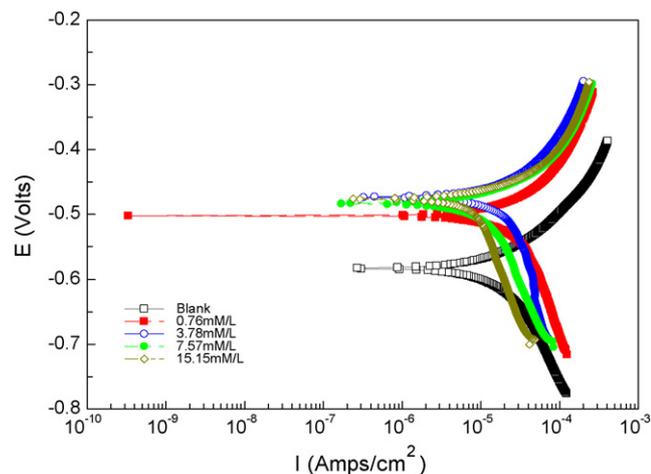

Fig. 2. Potentiodynamic polarization curves for steel immersed for 6 h in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde.

Fig. 3. Potentiodynamic polarization curves for steel immersed for 24 h in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde.
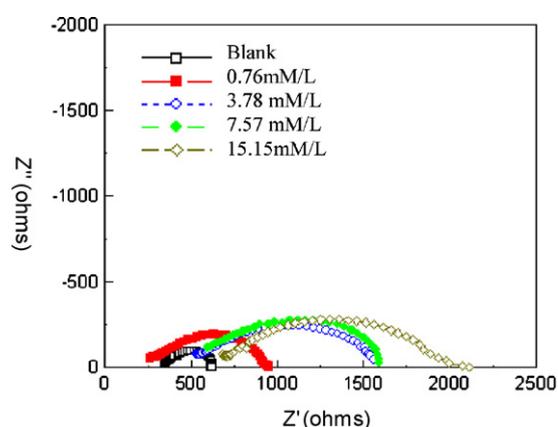

Fig. 4. Impedance diagrams for steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde (initial).

in the solution. The extrapolation of Tafel straight lines allows the calculation of the corrosion current (I_{corr}). The values of I_{corr}, the corrosion potential (E_{corr}), Tafel slopes (b_c) and (b_a) and percentage inhibition efficiency are given in Table 3. From the table, it can be seen that corrosion current decreases with increase of immersion time and the inhibition efficiency decreases from initial to 6 h duration, but increases for 24 h duration of immersion time.
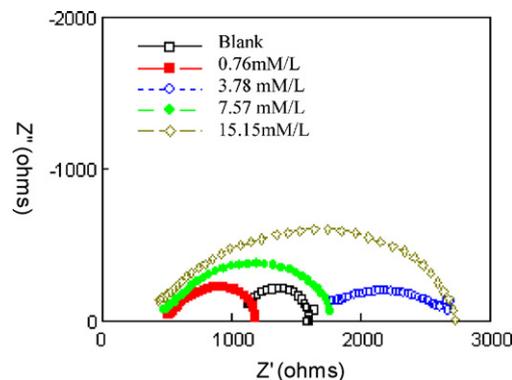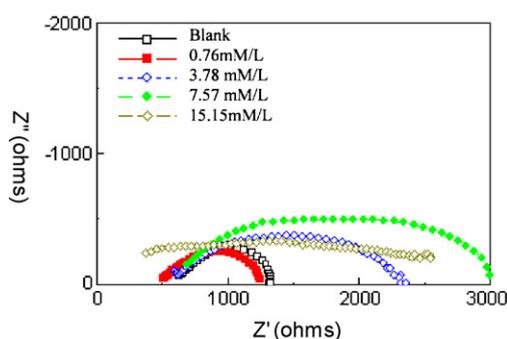

Fig. 5. Impedance diagrams for steel immersed for 6 h in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde.

Table 3
Potentiodynamic polarization parameters for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde for different duration of immersion at 30 °C

Inhibitor concentration (mM L^{-1})	E_{corr} (V vs. SCE)	I_{corr} (A cm^{-2})	Tafel slope (mV decade^{-1})		Corrosion rate (mmpy)	Inhibition efficiency (%)
			ba	bc		
Blank						
Initial	−0.5929	5.7219×10^{-5}	182	271	0.66	–
After 6 h	−0.5630	2.1201×10^{-5}	127	220	0.25	–
After 24 h	−0.5524	1.3133×10^{-5}	89	152	0.15	–
0.76 mM L^{-1}						
Initial	−0.5125	2.7812×10^{-5}	147	212	0.32	51.4
After 6 h	−0.5005	1.7625×10^{-5}	104	183	0.20	16.9
After 24 h	−0.4777	9.4040×10^{-6}	66	127	0.11	28.4
3.78 mM L^{-1}						
Initial	−0.4595	1.4370×10^{-5}	213	215	0.17	74.9
After 6 h	−0.4717	1.2034×10^{-5}	90	171	0.13	43.2
After 24 h	−0.4893	5.0618×10^{-6}	119	108	0.06	61.5
7.57 mM L^{-1}						
Initial	−0.4778	1.3203×10^{-5}	83	159	0.15	76.9
After 6 h	−0.4811	6.4858×0^{-6}	52	143	0.08	69.4
After 24 h	−0.4638	4.6571×10^{-6}	68	176	0.05	64.5
15.15 mM L^{-1}						
Initial	−0.4516	7.8804×10^{-6}	68	176	0.09	86.5
After 6 h	−0.4797	6.8679×10^{-6}	59	225	0.08	67.6
After 24 h	−0.4559	2.6748×10^{-6}	88	149	0.03	79.5

Fig. 6. Impedance diagrams for steel immersed for 24 h in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde.
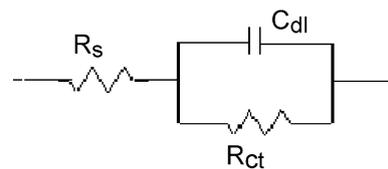
Table 4
Impedance parameters for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde at 30 °C

Inhibitor concentration (mM L^{-1})	R_t (Ω cm^2)	C_{dl} (F cm^{-2})	Inhibition efficiency (%)
Blank			
Initial	272	3.74×10^{-5}	–
After 6 h	618	1.77×10^{-7}	–
After 24 h	715	5.90×10^{-5}	–
0.76 mM L^{-1}			
Initial	710	1.51×10^{-5}	61.7
After 6 h	728	2.05×10^{-5}	15.1
After 24 h	794	2.05×10^{-5}	10.0
3.78 mM L^{-1}			
Initial	1089	2.02×10^{-5}	75.0
After 6 h	1274	4.99×10^{-5}	51.5
After 24 h	1833	6.12×10^{-6}	60.9
7.57 mM L^{-1}			
Initial	1149	2.95×10^{-6}	76.0
After 6 h	1401	6.51×10^{-6}	56.1
After 24 h	2703	2.16×10^{-6}	73.6
15.15 mM L^{-1}			
Initial	1381	1.73×10^{-5}	80.3
After 6 h	2467	2.30×10^{-6}	75.0
After 24 h	3841	7.10×10^{-8}	81.4

The addition of cinnamaldehyde in 4.76 mM L^{-1} of citric acid shifts E_{corr} to positive side indicating that the inhibition of corrosion of carbon steel in 4.76 mM L^{-1} of citric acid was under anodic control [35]. The corrosion current (I_{corr}) decreased with inhibitor concentration from 0.76 mM L^{-1} to. 15.15 mM L^{-1}. Maximum decrease in I_{corr} was obtained at 15.15 mM L^{-1}. The results obtained from the potentiodynamic polarization technique are in good agreement with those obtained from weight loss measurements.

3.2.2. Electrochemical impedance spectroscopy (EIS)

Figs. 4–6 show the Nyquist plots for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde obtained initially and for 6 h and 24 h of immersion in the solution. Impedance parameters such as charge transfer resistance (R_t), double layer capacitance (C_{dl}) and inhibition efficiency are given in Table 4. The following equivalent circuit was used [36] for analyzing the impedance data.

Equivalent circuit for impedance analysis

Rs = solution resistance , Rc$_{t=}$ Charge transfer resistance,

(C_{dl}) = double layer capacitance

From the table, it can be seen that the inhibition efficiency is decreased from initial to 6 h duration, but increases for 24 h duration of immersion time. R_t values increase upon prolonged exposure for 6 h and 24 h. This suggests that the product of reaction between carbon steel and test solution forms a film on the steel surface over the period of exposure and the rate of attack is lowered. On the metal surface free of any adsorbed layer, the dissolution in

acid media proceeds through consecutive or BDD mechanism [37]. This can be written as

$$FeH_2O_{ads} \rightleftarrows FeOH_{ads} + H^+ + e^- \qquad (7)$$

$$FeOH_{ads} \rightarrow FeOH^+ + e^- \qquad (8)$$

$$FeOH^+ + H^+ \rightleftarrows Fe^{+2} + H_2O \qquad (9)$$

The increase in the charge transfer resistance with time indicates that dissolution rate of carbon steel decreases with time.

The maximum inhibition efficiency (%) was found to be 80.3%, 75.0% and 81.4% for initial, 6 h and 24 h immersion, respectively, at 15.15 mM L^{-1} of cinnamaldehyde. This is due to increasing surface coverage by inhibitor which leads to an increase in inhibition efficiency with increasing inhibitor concentration. Thus increase in inhibition efficiency with the concentration indicates that cinnamaldehyde acts as an adsorption inhibitor. C_{dl} values are decreased with concentration of cinnamaldehyde. The decrease of capacitance with increasing inhibitor concentration may be
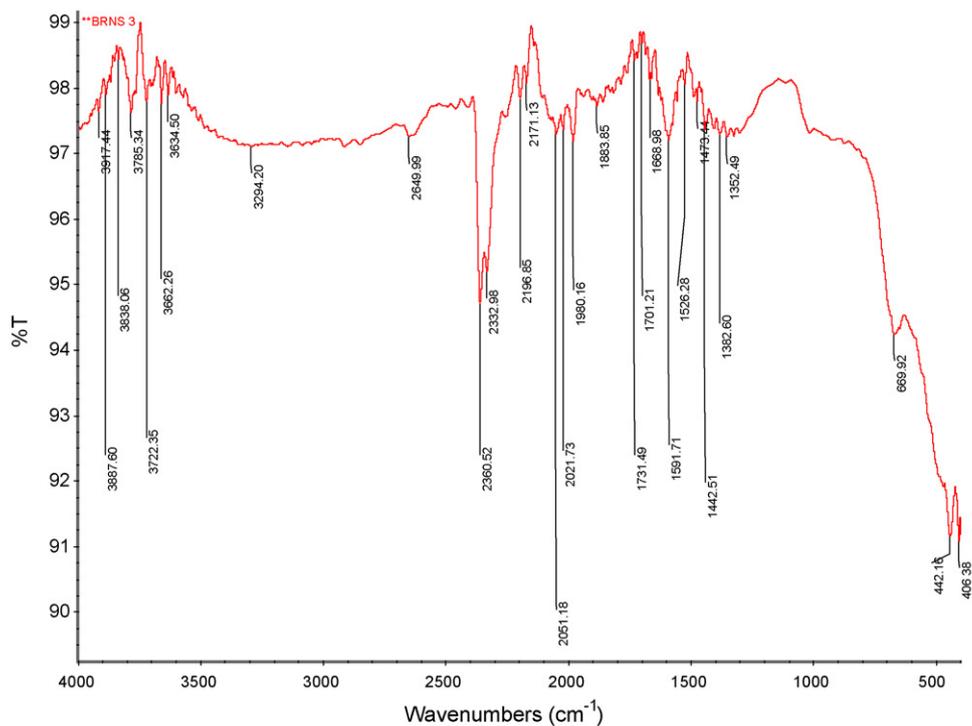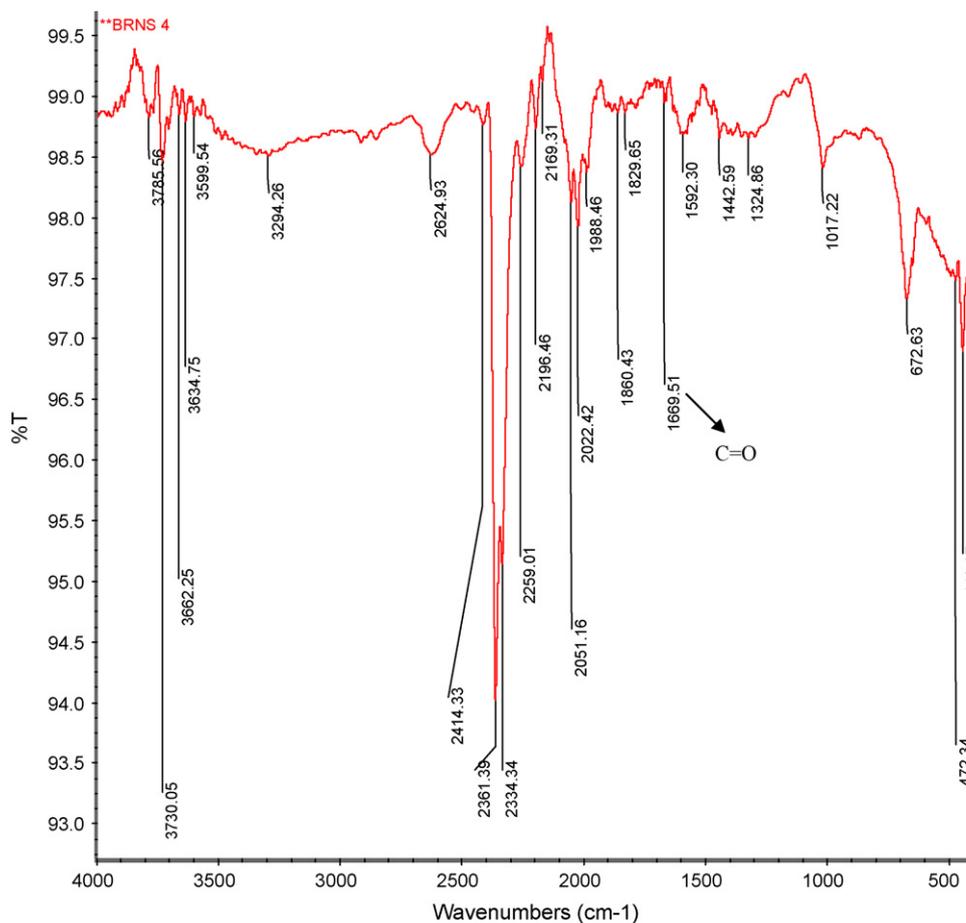

(a) FTIR spectrum of carbon steel immersed in 4.76 mM/L of citric acid for 24h at 30 °C.
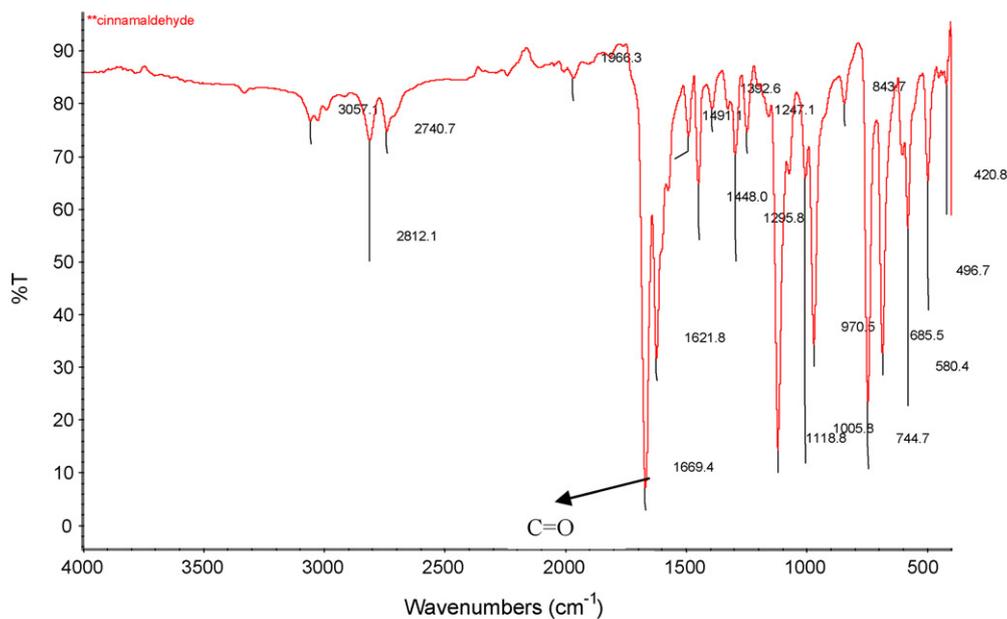
(b). FTIR spectrum of cinnamaldehyde.

Fig. 7. (a) FTIR spectrum of carbon steel immersed in 4.76 mM L^{-1} of citric acid for 24 h at 30 °C. (b). FTIR spectrum of cinnamaldehyde. (c) FTIR spectrum of carbon steel immersed in 4.76 mM L^{-1} of citric acid containing 15.15 mM L^{-1} of cinnamaldehyde for 24 h at 30 °C.

(c) FTIR spectrum of carbon steel immersed in 4.76 mM/L of citric acid containing 15.15 mM/L of cinnamaldehyde for 24 h at 30°C.

Fig. 7. (*Continued*).

attributed to a decrease in local dielectric constant and an increase in the thickness of the electrical double layer, [38] suggesting that cinnamaldehyde functions by adsorption at the solution interface. The inhibition efficiencies calculated from AC impedance results also show the same trend as those obtained from dc polarization and weight loss measurements.

3.3. Atomic absorption spectrophotometric studies

Percentage inhibition efficiency of the inhibitor towards the dissolution of iron was calculated and the results are given in Table 5.

Table 5
Amount of iron content and inhibition efficiency obtained for carbon steel immersed in 4.76 mM L^{-1} of citric acid containing different concentrations of cinnamaldehyde for different durations of immersion at 30°C by atomic absorption spectroscopy method

Inhibitor concentration (mM L^{-1})	Amount of iron content (mg L^{-1})		Inhibition efficiency (%)	
	6 h	24 h	6 h	24 h
Blank	96.9	30.0	–	–
0.76	81.4	29.8	16.0	0.66
3.78	40.4	18.6	58.3	38.0
7.57	33.7	17.6	65.2	41.3
15.15	28.3	10.8	70.8	64.0

The percentage inhibition efficiency obtained by this technique was found to be in good agreement with that obtained from the conventional weight loss method.

3.4. Surface examination studies FTIR spectral studies

The FTIR spectrum obtained for the carbon steel immersed in 4.76 mM L^{-1} of citric acid for 24 h is shown in Fig. 7a. The FTIR spectrum of cinnamaldehyde is also taken and given in Fig. 7b. Fig. 7c shows the FTIR spectrum of carbon steel immersed in 4.76 mM L^{-1} of citric acid containing an optimum concentration (15.15 mM L^{-1}) of cinnamaldehyde. It is clear from Fig. 7b, that peaks corresponding to 〉C=O stretching frequency of aldehyde group is observed at 1669 cm^{-1}. On comparing Fig. 7c with Fig. 7b, it is observed that the peaks characteristic of cinnamaldehyde is observed in Fig. 7c also. Further the intensity of the C=O stretching frequency is decreased in Fig. 7c which implies that the oxygen atom of cinnamaldehyde is coordinated to Fe^{2+} resulting in the formation of a Fe^{2+}–inhibitor complex on the metal surface.

4. Conclusions

Inhibition efficiency increases with increase in concentration of cinnamaldehyde. The results obtained from electrochemical stud-

ies are in good agreement with those obtained from weight loss measurements. The increase of charge transfer resistance with time indicates that dissolution rate of carbon steel decreases with time. The formation of the adsorbed protective inhibitor film on the carbon steel was also confirmed by FTIR studies.

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